MAHARSHI DAYANAND SARASWATI UNIVERSITY

AJMER

Format for Outline of Proposed Research work

1. **Name of Scholar** (In English): **MONIKA DADHEECH**

(In Hindi): मोनिका दाधीच

2. Title of the Research Work:

(In English): Photooxidation of Some Industrially Important Organic Compounds by Strontium Chromate

(In Hindi): कुछ औद्योगिक महत्व के कार्बनिक यौगिकों का स्ट्रॉन्शियम क्रोमेट द्वारा प्रकाशीय ऑक्सीकरण

3. Location:

a. Institutaion /Department where the work is to be done

Department of Chemistry,
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b. Geographical area of Investigation ,if any. **N/A**

4. Introduction,Importance of proposed Investigation

A wide variety of organic contaminants are introduced into the water resources from industrial effluents, agricultural runoff and chemical spills. Their toxicity and stability to natural decomposition and persistence in the environment has been the cause of much concern to the societies and regulatory authorities around the world. Thus, development of some appropriate method for the degradation of various toxic or non biodegradable compounds seems necessary present in drinking ground, surface waters and waste waters.

The biggest consumers of water is textiles, tannery, electroplating, pulp and paper industries and therefore, these industries are also considered mostly responsible for creating water pollution. Photo catalytic degradation has emerged as an efficient process for degradation of organic pollutants, which are present in the effluents released by these industries.

Most synthetic industrial dyes are complex aromatic compounds with an azo bond connected to several aromatic structures. Some, however, are polymeric structures containing metals. It is estimated that there are over 10,000 commercially available dyes and pigments for industrial use. The annual consumption is around 7×10^5 tons worldwide [1, 2]. However, about 10 ~ 15% of the synthetic dyes produced are discharged into industrial effluents, causing environmental problems. Accordingly, in many countries the contamination of water bodies by dyes is an important problem. The colour of textile effluents is due to the dyes. Synthetic dyes vary in chemical composition, but they share a common features like they must be highly stable against light, washing and they must be resistant to chemical and microbial attack[3]. The chief pollutants are chlorinated organic derivatives, chlorinated solvents, nitro derivatives of

benzene and phenol, pesticides (triazine derivatives) and azodye derivatives. Among the chemical classes of dyes, azo dyes, the largest class of these compounds (84% of all dyes used), are considered to be recalcitrant, non-biodegradable by aerobic bacteria, and persistent. The anaerobic process has a major disadvantage in that the reduction of azo dyes generates colorless but potentially mutagenic and carcinogenic aromatic amines[4]. Consequently, the treatment of dye-polluted effluents is considered to be one of the most challenging tasks among the environmental community. Many treatment methods have been reported for dye degradation such as flocculation, coagulation[5], ultrafilteration[6], reverse osmosis[7], biodegradeation[8] etc. These methods have their own limitation in terms of high cost, specificity for certain group, disposal of sludge formed, and excessive use of chemicals. All these result in secondary pollution[3]. However, none of these methods has been found to be very suitable.

On the contrary, bleached dye after degradation of solution is relatively less toxic and almost harmless. Secondly, dye containing colored water is of almost no practical use, but if this colored solution is bleached to give colorless water, then it may be used for some useful purposes like washing, cooling, irrigation and cleaning. The photocatalytic bleaching or degradation seems to be quite promising as it can provide a low cost method to solve this problem. Effectively remove dyes from effluent hence study of alternative treatment technologies is necessary. Photocatalytic degradation provides an alternative method for waste water treatment in low cost, less time and easy operative method. Photocatalytic degradation being eco friendly and cost effective is a preferred method.

Most of these compounds are not degraded by direct exposure to sun light since there absorption rate in the visible range is very less. Moreover majority of these compounds are stable and can persist for sufficiently long time. One of the technique of removal of these contaminates is by degradation of dyes by using semiconductor and mixed oxide photo catalysts (such as TiO_2, ZnO, Cds, $SrCrO_4$). But most of these photo catalysts work under UV light, which restricts their applications in sunlight since sunlight that reaches the earth surface contain only 4% UV light. There is few catalyst that are active under visible light, but their efficiency is low due to fast charge recombination. Hence, the main challenge among the scientists is to search newer materials to reduce that recombination time [9-11]. Among these materials, $SrCrO_4$ with a band gape of 2.8eV is an attractive candidate for photo catalysis.

So a ternary semiconductor i.e. strontium chromate is successfully used as a photo catalyst for degradation of some industrially important organic compounds, which will explore its use for removal of a variety of industrial effluents in future.

Objective-

(i) To convert harmful dyestuff into non hazardous products using cheapest energy source sunlight.

(ii) To discover a new catalyst this is active in Visible light.

(iii) Introduce a catalyst to increase rate of photo oxidation reaction of organic compound by reduce recombination time.

(iv) To develop a pathway of photo oxidation of industrial effluents by $SrCrO_4$.

5. Review (Work already done on the subject of proposed Research Study.)

It's estimated that about 20% of dyes are lost throughout the dyeing Process are discharged directly into the environment [Hussein[12], Madhavan et al.[13],] . More than 50% of textile dyes in azoic dyes which are recognized by nitrogen π-bond [Song et al.[14]]. Textile and industrial dye contain large group of organic compound that produce more than 7, 00,000 ton per year.

During the last decade, photocatalysts have been developed and applied to the removal of dyes from industrial effluents[15]. Various support materials (silica, alumina, zeolites or clays[16]) and coating methods have been proposed for degradation of several organic compounds[17].

E. Samsudin et al.[18] has investigated the photocatalytic degradation of Reactive Blue 4 (RB4), an anthraquinone dye, by using pure anatase nano titanium (IV) oxide (TiO_2). The dye molecules were fully degraded and the addition of hydrogen peroxide (H_2O_2) enhanced the photodegradation efficiency. It is found that the degradation as the hydroxyl radicals in the bulk solution is sufficient for complete mineralization. The disappearance of the dye follows pseudo-first-order kinetics.

K. Pamecha et al.[19] studied efficiency of $CeFeO_3$ as photocatalyst for degradation of Coralene Dark red 2B (Disperse red 167). Photocatalytic bleaching of Coralene Dark red 2B was observed spectrophotometrically. The influence of variation of different reaction parameters such as concentration of

dye, pH, and amount of photocatalyst were observed. The optimum conditions for photocatalytic degradation have been established. The kinetics of degradation of the dye follows first order kinetics. A tentative mechanism for degradation of dye by $CeFeO_3$ has been proposed. The presence of hydroxyl radical as an active oxidizing species has been confirmed.

Khalik et al.[20] worked on the effects of number of sulphonic groups on solar photocatalytic degradation of Acid Orange 7 (AO7), Orange G (OG), New Coccine (NC) ,Reactive Black 5 (RB5) and Reactive Green 19 (RG19) in aqueous solution. The data obtained for photocatalytic degradation of AO7, OG, NC, RB5 and RG19 were well fitted with the Langmuir–Hinshelwood kinetic model. The pseudo-first-order rate constant of RG19 with six sulphonic groups was the highest among the dyes in this study. Results shown that photocatalytic degradation rate of the azo dyes increased following the increase of the number of sulphonic groups in the dyes in the sequence of: AO7 < OG < NC < RB5 < RG19.

T. Ghorai et al.[21] Focused on Nano-sphere TiO_2 and $SrCrO_4$ (up to 5 mol %). Mixed oxides were synthesized by co-precipitation method and investigated as catalyst in the photodegradation of rhodamine 6G (Rh6G) in aqueous solution. The results shown that the mixed oxides of TiO_2 with 1 mole % of $SrCrO_4$ (SCT1) exhibit a photocatalytic activity 3 times higher than that of $P25TiO_2$ for oxidation of various dyes (Rh6G, TB, MO and BG) under UV light irradiation ($\lambda > 280$ nm).

U. Vyas et al.[22] carried out the photocatalytic degradation of xylidine ponceau out by using Well-Dawson catalyst in the presence of light. Well-

Dawson catalyst was synthesized by using cupric chloride dihydrate, chromium chloride and sodium tungstate. It has been utilized for degradation of xylidine ponceau dye. A 200 W tungsten lamp was used for irradiation. Progress of the reaction was followed kinetically by measuring absorbance of the reaction mixture at various time intervals.

Troupis et al.[23] carried out the photocatalytic reductive oxidative degradation of acid orange-7 by using polyoxometallate. While polyoxometallate based photocatalyst assembled with cucurbit [6] uril-via-hydrogen bonds for degradation of azo dyes was developed by Cao et al.[24].

Two new inorganic-organic hybrid compounds based on well-defined Keggin type polyoxometallate and copper-bis (triazole) assemblies namely have been synthesized by Wu et al.[25] while Wang and Yang[26] synthesized phosphotungustic and silicotungustic acid. They investigated their photocatalytic activity in the degradation of organic dye methyl orange.

The photocatalytic degradation of methylene blue by MoO_3 modified TiO_2 under visible light was carried out by Yang et al.[27] Wang and Wu[28] synthesized TiO_2 nanoparticles by a facile hydrothermal method. TiO_2 Nanoparticles exhibit much better photocatalytic activity towards the photodegradation of benzene.

Mirkhani et al.[29] carried out the photocatalytic degradation of some textile azo dyes by using TiO_2 and modified TiO_2 with Ag metal (1% w/w) in aqueous solution under irradiation with a 400 W high pressure mercury lamp. Ternary zinc spinal oxides such as Zn_2SnO_4, $ZnAl_2O_4$ and $ZnFe_2O_4$ were

synthesized and characterized, and their activities in the photodegradation of phenol molecules were investigated by Anchieta et al.[30]

The photocatalytic bleaching of methylene blue was carried out by Ameta et al.[31] in the presence of semiconducting iron (III) oxide. The degradation was observed spectrophotometrically. The effect of various operating variables like pH, concentration of dyes, amount of semiconductor, light intensity, particle size, stirring, etc. was observed on the rate of the reaction. Methylene blue (MB) degradation has also been investigated by Houas et al.[32] in aqueous heterogeneous suspensions. Removal of the colour by TiO_2/UVbased photocatalysis with simultaneous oxidation of the dye, with an almost complete mineralization of carbon, nitrogen and sulfur heteroatoms into corresponding counter parts CO_2, NH^{4+}, NO^- and SO_4^{2-}, respectively was observed.

Eskan et al.[33] synthesized cadmium sulphide nanostructures via a simple precipitation method. The photocatalytic activities of the synthesized samples were investigated for degradation of methylene blue (MB) under blue LED (3W) and solar light irradiation. Bi_2O_3 nanostructures with different dimensionalities including dots, rods and sheets have been synthesized by Huang et al.[34] for the study of their photocatalytic activities in degradation of organic dyes while Sharma et al.[35] used bismuth sulfide as photocatalyst for the degradation of rose Bengal.

The photocatalytic degradation of p-chlorophenol by H_2O_2/TiO_2 hybrid in aqueous suspension under irradiation was reported by Nguyen and Juang[36].

K. seyyedi et al.[37] investigated removing of C.I. Direct Black 38 by photocatalytic method as one of advanced oxidation processes (AOPs) using

TiO_2 as photocatalyst and determination of optimum values of effective parameters against color removing efficiency. Spectrophotometric results shown that photocatalytic process has decolorization ability of wastewaters containing C.I. Direct Black 38 in different dye concentrations.

K. Milenova et al.[38] determined that activated copper doped ZnO can be used as an effective photocatalyst for the remove of azo dye reactive black 5 in waste water under UV radiation. Photocatalytic activities of copper doped zinc oxides have been investigated for the degradation of Methylene Blue dye [39], resazurin dye [40] and Methyl Orange [41].

Barium Chromate powder is used as a photocatalyst to degrade Eosin yellow[42]. $BaCrO_4$ (also called Hashemite) with a band gap of 2.63 eV is an attractive candidate for photocatalysis[43].

Li et al., [44] investigated the photodegradation of methylene blue using the TiO_2/Ti electrode under different experimental conditions in presence of UV. Chemical oxygen demand (COD) removal was also studied. The prepared electrode mainly consisted of anatase TiO_2 nanoparticles on its surface and exhibited a superior photocatalytic activity. The COD removal during the photodegradation of methylene blue by TiO_2/Ti mesh electrode was 97.3%.

Pereira et al.,[45] studied that degradation of two xanthene dyes, Erythrosine B (Ery) and Eosin Y (Eos), using UV/TiO_2 in a photocatalytic reactor. Photocatalysis was able to degrade 98% of Ery and 73% of Eos and results in removal of 65% chemical oxygen demand. It was revealed that it is a pH dependent process and better results were obtained under acidic conditions.

Byberg et al.,[46] studied the photocatalytic degradation of a series of six acid dyes (Direct red 80, Direct red 81, Direct red 23, Direct violet 51, Direct yellow 27 and Direct yellow 50), which was compared in terms of color removal, mineralization and toxicity (Lactuca sativa L. test) after photocatalysis on immobilized titanium dioxide.

Bi_2O_3-Ruduced graphene oxide (RGO) composite with 2 wt.% RGO achieves maximum methyal orange and methylene blue degradation rates of 93% and 96% at 240 min. under visible light irradiation. The enhanced photocatalytic performance is ascribed to the increased light adsorption and the reduction in electron-hole pair recombination in Bi_2O_3 with the introduction of RGO by Liu X[47].

Photocatalytic bleaching of hydrolyzed textile azo dye Direct Crysophenine G dioxide using titanium (TiO_2) was analyzed by P Mehta et al.,[48] The Kinetic analysis of photocatalytic bleaching reveals that the degradation follows approximately pseudo first order kinetics according to the Longmuir- Hinshelwood model. The effect of addition of transition metal ions (Fe^{2+}, Cu^{2+}, Mn^{2+}, Zn^{2+}, Ag^{+}) on photo bleaching efficiency of TiO_2 investigated.

A study done by M Surana et al., [49] revealed that the Photo Fenton reagent can be used to decolorization and minralization of azo dye Reactive Yellow 86. The efficiency of treatment was evaluated by color alteration and minralization of the dye.

Decolourization and mineralization of Commercial Textile Dye Acid Red 18 by Photo-Fenton Reagent and study of effect of homogeneous catalyst Uranyl Acetate developed by M Surana et al.[50].

P Mehta et al.,[51] studied that the Photocatalytic degradation of a textile azo dye, Acid Orange 8 in aqueous solution under visible light using TiO_2 as catalyst. Kinetic analysis of photodegradation reveals that the degradation follows first order reaction kinetics. The effect of nature of various semiconductor catalysts on the degradation process is also monitored and it is found that the rate of photobleaching of dye in visible light is decreased as the band gap energy of semiconductor catalyst is increased.

The photocatalytic degradation of synthetic dye solution of reactive blue 2 was investigated by Rashidi et al.[52] using UV irradiation in aqueous suspension of nanotitania as photocatalyst and H_2O_2 as electron acceptor in a slurry photoreactor.

Photocatalytic degradation of Methyl orange and Methylene blue has been carried out by heterogeneous photocatalytic process using TiO_2 as semiconductor by Mehra and Sharma[53]. The effect of process parameters like concentration of dye and pH on photocatalytic degradation of Methyl orange and Methylene blue was observed.

6. Research Gap identified in the proposed field of investigation. (Based on review)-

The degradation of industrial and textile organic compound done by photochemistry already reported in the literature. It is concerned with different method and catalysts. But photo oxidation is booming field of chemistry due to its numerous potential applications. Using light as an energy source to drive a reaction in a desired direction has result in chemistry which is greener.

Most of the Photocatalyst work under UV light. Only 4% UV light reaches on earth's surface by sun light. Hence need a catalyst for reduce the recombination time which is active in Visible light, so work on $SrCrO_4$.

7. Major Hypothesis -

(i) Assuming that the rate of Photo oxidation reaction will be fast by $SrCrO_4$ due to low band gap.

(ii) Assuming that it will provide a new approach in Chromate Photo chemical reaction.

This is Heterogeneous photocatalysis. It is a technology based on the irradiation of a catalyst, usually semiconductor, which may be photo excited to form donor site (reducing site) and electron acceptor site (oxidizing site).

$SrCrO_4$ has an appropriate energy separation between its valance band (VB) and conduction band (CB), which can be surpassed by the energy of solar photon. The band gape energy is 2.8eV. The first event, after absorption of UV/VIS radiation, the generation of electron / hole pairs separated between CB and VB. These holes are highly oxidizing and the corresponding photoelectrons

are having sufficient electro negativity to reduce dioxygen to superoxide / hydroperoxide radicals. These e^-/h^+ may lead to complete mineralization of organic compounds.

8. Research Methodology-

(1) Selection of Substrate: - The substrate selected for the present study some industrially important organic compounds like Dyes which are used in the industries mostly azo dyes like acid orange-7, methylene blue, Acid Red 18, C.I. Direct Black 38, reactive black 5 and other related organic compound.

(2) Exposure to light:- The solution of industrially important organic compound and Strontium chromate will be irradiated with visible light (Tungsten lamps, 2X 200W, Philips).

(3) Progress of the reaction:- The progress of the reaction will be checked by TLC, qualitative and spectrophotometric analysis at regular time intervals.

(4) Control Experiments:- The control experiments will be also performed in the following manner-

(i) Substrate, Strontium chromate and oxygen in light.

(ii) Substrate, light and oxygen (No strontium chromate will be added).

(iii) Substrate, strontium chromate and light (No oxygen will be bubbled).

(5) Separation and Characterization of the Photoproduct: - The photoproduct of the reaction will be separated by the preparative thin layer chromatography and chemical methods. The characterization of the photoproduct will be done by different physical, chemical and spectral method.

A probable mechanism for photo-oxidation of each substrate will be proposed.

Experimental-

Following procedure will be adopted for the investigation:

(i) All the chemical used will be of Analar grade, all solvent will be distilled before use.

(ii) Photo-oxidation will be carried out in borosil glassware's, with dried equipments.

iii) Silica gel- G (Merck) will be used for thin layer chromatography. Glass plates (7.5 X 2.5 cm.) will be used for making TLC plates.

(iv) Air bubbled in the reaction mixture will be passed with the help of an aerator.

(v) Tungsten lamps (2 X 200 W, Philips) will be used for irradiation purpose. At proper distance between lamp and reaction vessel. Water cells (15 cm) will be placed between light source and reaction vessel to eliminate heat radiations.

(vi) All melting points will be determined on a Toshniwal apparatus.

(vii) The pH of the reaction mixture will be checked by systronics- 327 griph (digital) pH meter.

(viii) Elemental analysis will be done using Carlo-Erba-1106 automatic analyzer.

(ix) Spectrophotometric studies will be performed on a Schimadzu UV-1700 Pharmaspec spectrophotometer.

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मोनिका पाठीच

Signature of the Candidate with date - 28-5-2016

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